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Indian Standard
SPECIFICATION FOR BITUMEN-BASED FILLING COMPOUNDS FOR ELECTRICAL PURPOSES

(Fourth Reprint MARCH 2001)

UDC 621.315.616.97 : 678.584

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 7 *February* 1974

AMENDMENT NO. 1 FEBRUARY 1979
TO
IS : 7084-1973 SPECIFICATION FOR BITUMEN-BASED FILLING COMPOUNDS FOR ELECTRICAL PURPOSES

[This standard covers the requirements of bitumen-based compounds suitable for use as filling compounds only. The object of this amendment is to include in this standard the requirements of bitumen-based compounds for use as impregnating compounds.]

Alterations

(*First cover page, pages* 1 *and* 3, *title*) — Substitute the following for the existing title:

'Indian Standard

SPECIFICATION FOR BITUMEN-BASED
COMPOUNDS FOR ELECTRICAL PURPOSES'

(*Page* 3, *clause* 1.1, *line* 2) — Substitute 'filling and impregnating' *for* 'filling'.

(*Page* 4, *Table* 1) — Substitute the following for the existing table:

TABLE 1 CLASSIFICATION

(*Clause* 2.1)

TYPE OF COMPOUND	DESCRIPTION	CLASS	SOFTENING POINT	
			Minimum	Maximum
(1)	(2)	(3)	(4)	(5)
Filling compounds	Asphaltic bitumen base compound	I	—	40°C
		II	Over 40°C	70°C
		III	Over 70°C	90°C
		IV	Over 90°C	120°C
		V	Over 120°C	—
	Natural asphalt base and/or asphaltic bitumen base compounds containing mineral matter	VI	Subject to agreement between the purchaser and the supplier	
Impregnating compound	Bitumen base compound	VII	Over 70°C	90°C

(*Page* 4, *clause* 4.2, *line* 2) — Substitute 'Classes II, III, IV, V, VI and VII' *for* 'Classes II, III, IV, V and VI'.

(*Page* 4, *clause* 6.1, *line* 1) — Substitute 'Classes II, III, IV, V, VI and VII' *for* 'Classes II, III, IV, V and VI'.

(*Page* 7, *clause* 15.1, *line* 1) — Substitute 'Classes II to VII' *for* 'Classes II to VI'.

(*Page* 21, *clause* D-4, *heading*) — Substitute '**CLASSES I TO V AND VII**' *for* '**CLASSES I TO V**'.

(*Page* 24, *clause* G-1.1, *lines* 5 *and* 6) — Substitute 'Classes I, II, III, IV or VII' *for* 'Classes I, II, III or IV'.

Addenda

(*Page* 5, *Table* 2, *Class* VI) — Add the following new matter against respective entries after 'Class VI':

Sl No.	Property	Class VII
1	Softening point:	As agreed to between manufacturer and purchaser
	Tolerance on the agreed value	—
2	Penetration at 27°C (*Min*)	26
3	Electric strength (proof) test:	
	Test voltage, kV (rms)	20
	Test temperature, °C	60 ± 1.
4	Mineral matter (ash)	<0·5%
5	Contraction and depth of pipe:	
	Contraction	<6·5%
	Depth of pipe	<18·5 mm
6	Solubility (insoluble content, *Max*)	0·5%

(*Page* 7, *clause* 13.1, *Note*) — Add the following new matter at the end of the Note:

'The coefficient of expansion for compound of Class VII is under consideration.'

(*Page* 28, *Table* 4, *Class* VI) — Add the following new matter under the respective columns after Class VI:

'Class	Mould Temperature
VII	Softening point'

(ETDC 18)

Printed at New India Printing Press, Khurja, India

Indian Standard
SPECIFICATION FOR BITUMEN-BASED FILLING COMPOUNDS FOR ELECTRICAL PURPOSES

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(*Continued on page* 2)

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(*Continued from page* 1)

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Indian Standard
SPECIFICATION FOR BITUMEN-BASED FILLING COMPOUNDS FOR ELECTRICAL PURPOSES

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 25 April 1973, after the draft finalized by the Insulating Materials Sectional Committee had been approved by the Electrotechnical Division Council.

0.2 Bitumen-based filling compounds are being used as filling material for cable jointing, busbar filling, impregnating, insulating tapes, etc. The manufacture of such type of compound having been well established in this country, the need was felt for adopting uniform specification for these compounds. This Indian Standard has been prepared to meet this need.

0.3 In preparing this Indian Standard assistance has been drawn from Document No. 68/30345 ' Draft British Standard specification for bitumen-base filling compounds for electrical purposes ' (revision of BS : 1858) issued by the British Standards Institution.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard covers compounds of bituminous nature suitable for use as filling compounds for electrical purposes.

2. CLASSIFICATION

2.1 The compounds covered by this standard are classified as given in Table 1.

3. GENERAL

3.1 The compound shall be stable and shall neither froth nor emit excessive fumes when heated to a temperature of 80°C above the softening point.

---

*Rules for rounding off numerical values (*revised*).

TABLE 1 CLASSIFICATION

(*Clause* 2.1)

Description	Class	Softening Point	
		Minimum	Maximum
Asphaltic bitumen base compound	I	—	40°C
	II	Over 40°C	70°C
	III	Over 70°C	90°C
	IV	Over 90°C	120°C
	V	Over 120°C	—
Natural asphalt base and/or asphaltic bitumen base compounds containing mineral matter	VI	Subject to agreement between the purchaser and the supplier	

3.2 Under normal conditions of use there shall be no formation of gas pockets, cavities or cracks in the compound on cooling.

3.3 The compound shall not have any injurious effect on copper, brass, iron, lead or aluminium, zinc, galvanized steel and their alloys at normal temperature of installation and operation.

3.4 The compound shall be capable of being remelted repeatedly without significant separation of the constituents.

4. SOFTENING POINT

4.1 The softening point shall be determined by the 'ring and ball' method as described in IS : 1205-1958*.

4.2 The softening point of Class I compound shall not exceed 40°C and the softening points of Classes II, III, IV, V and VI compounds shall be the agreed values subject to the tolerances given in Table 2.

5. SPECIFIC GRAVITY

5.1 The specific gravity of the compound, when determined by a method given in IS : 1202-1958† shall lie within 1·0 to 1·05 at 27 ± 2°C.

6. PENETRATION

6.1 The penetration value of compounds of Classes II, III, IV, V and VI shall be the subject of agreement between the purchaser and the manufacturer, provided that it is not less than the appropriate value given in Table 2 and that it does not vary from the agreed value by more than the

---

*Methods for testing tar and bitumen: Determination of softening point.

†Methods for testing tar and bitumen: Determination of specific gravity.

TABLE 2 · PROPERTIES OF BITUMEN-BASED FILLING COMPOUNDS

(*Clauses* 4.2, 6.1, 10.1, 13.1, 13.2 *and* 14.1)

Sl No.	Property	Class I	Class II	Class III	Class IV	Class V	Class VI	Method of Test, Ref to
1	Softening point	Shall not exceed 40°C	As agreed to between manufacturer and purchaser					IS : 1205-1958*
	Tolerance on the agreed value	—	± 3°C	± 3°C	± 5°C	± 5°C	± 3°C	
2	Penetration at 27°C (*Min*)	—	20	20	10	7	7	IS : 1203-1958†
3	Electric strength (proof) test:							Appendix B
	Test voltage, kV (rms)	15	20	20	20	20	15	
	Test temperature, °C	60 ± 1	60 ± 1	60 ± 1	90 ± 1	90 ± 1	60 ± 1	
4	Mineral matter (ash)	< 0·5%	< 0·5%	< 0·5%	< 0·5%	< 0·5%	As agreed to between the purchaser and the supplier but not exceeding 37 percent	Appendix D
5	Contraction and depth of pipe:							Appendix G
	Contraction	< 8%	< 8%	< 8%	Shall be the subject of agreement between the purchaser and the supplier		6·5 percent	
	Depth of pipe	< 25 mm	< 25mm	< 25 mm	do		< 25 mm	
6	Solubility (insoluble Content, *Max*)	0·5%	0·5%	0·5%	0·5%	0·5%	40%	IS : 1216-1958‡ (Method A for Classes I, II, III, IV, V and Method B for Class VI)

*Methods for testing tar and bitumen: Determination of softening point.

†Methods for testing tar and bitumen: Determination of penetration.

‡Methods for testing tar and bitumen: Determination of solubility in carbon disulphide.

tolerance given in Table 3. The method of test and other details shall be in accordance with IS : 1203-1958*.

TABLE 3 TOLERANCE ON PENETRATION

(*Clause* 6.1)

AGREED VALUE	TOLERANCE
Up to 10	± 2
11 to 80	± 4
81 and above	± 10

7. POURING POINT

7.1 The pouring point of compounds of Classes II, III, IV, V and VI shall be the subject of agreement between the purchaser and the manufacturer and shall be determined as described in Appendix A. The tolerance on pouring point shall be $^{+5}_{-0}$°C.

8. ELECTRIC STRENGTH (PROOF) TEST

8.1 Two samples of the compound shall be tested as described in Appendix B. If one sample fails to withstand the appropriate voltage, three additional samples should be tested. If any sample fails in the additional test, the result shall indicate failure to comply with the standard, but if all the additional samples pass the test, the result shall indicate the compliance with the standard.

9. FLASH POINT

9.1 The flash point of the compound of any class, when determined by either of the methods described in Appendix C shall not be less than 200°C. In case of dispute, Method C of IS: 1209-1958† using the Pensky-Martens apparatus shall be used.

10. MINERAL MATTER

10.1 The amount of mineral matter (ash) in the compound as determined by the method described in Appendix D shall not exceed the appropriate value given in Table 2.

11. FREEDOM FROM SULPHUR

11.1 The compound shall be tested as described in Appendix E and there shall be not more than a slight discoloration of the copper foil.

---

*Methods for testing tar and bitumen: Determination of penetration.

†Methods for testing tar and bitumen: Determination of flash point and fire point.

12. FREEDOM FROM ACIDITY

12.1 When tested in accordance with the method described in Appendix F, not more than 4 mg of potassium hydroxide shall be required to neutralise 1 g of compound.

13. CONTRACTION AND DEPTH OF PIPE

13.1 Contraction — The contraction of the compound shall not exceed the appropriate value given in Table 2 when tested in accordance with the method described in Appendix G.

NOTE — The coefficient of expansion of compounds of Classes I to V is of the order of 0·000 65 per Celsius degree on the volume at 20°C and for Class VI is of the order of 0·000 55 per Celsius degree.

13.2 Depth of Pipe — The depth of pipe shall not exceed the appropriate value given in Table 2, when tested as described in Appendix G.

NOTE 1 — This test is useful in comparing the behaviour, on cooling, of compounds with similar softening points and eliminates those which by virtue of their composition have a sharper softening point and a tendency to excessive pipe formation.

NOTE 2 — In the practical use of compounds, the extent of pipe or depression formation is chiefly governed by the pouring temperature and conditions of cooling. Pipe formation may be reduced by taking care to select a compound having the lowest softening point and pouring temperature consistent with the purpose for which the compound is required, and by encouraging cooling from below upwards by shielding the top portion of the apparatus during the cooling process.

14. SOLUBILITY

14.1 The solubility of the compound shall be determined by the methods described in IS :1216-1958*. The insoluble content shall not exceed the appropriate value given in Table 2.

15. ADHESIVENESS

15.1 The adhesiveness of the compounds of Classes II to VI shall be determined by the method described in Appendix H. Adhesion of the compound of any class to the base plate shall occur over an area of not less than 90 percent of that part of the plate which has been exposed to the molten compound.

16. PACKING AND MARKING

16.1 Packing — The material shall be packed in suitable containers preferably in following sizes:

1, 5, 7, 10, 20 and 50 kg.

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*Methods for testing tar and bitumen: Determination of solubility in carbon disulphide.

16.2 Marking — The container shall be indelibly marked with the following information:

- a) Manufacturer's name or trade-mark or both,
- b) Class of compound,
- c) Batch number,
- d) Quantity of compound,
- e) Date of packing, and
- f) Reference to this Indian Standard.

16.2.1 The containers may also be marked with the ISI Certification

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

APPENDIX A

(*Clause* 7.1)

DETERMINATION OF POURING POINT OF COMPOUNDS OF CLASSES II TO VI

A-1. INTRODUCTION

A-1.1 The pouring point is the temperature at which 50 ml of compound flows out of the standard apparatus in 25 seconds.

NOTE — This is equivalent for all practical purposes to a viscosity of 1 000 centistokes.

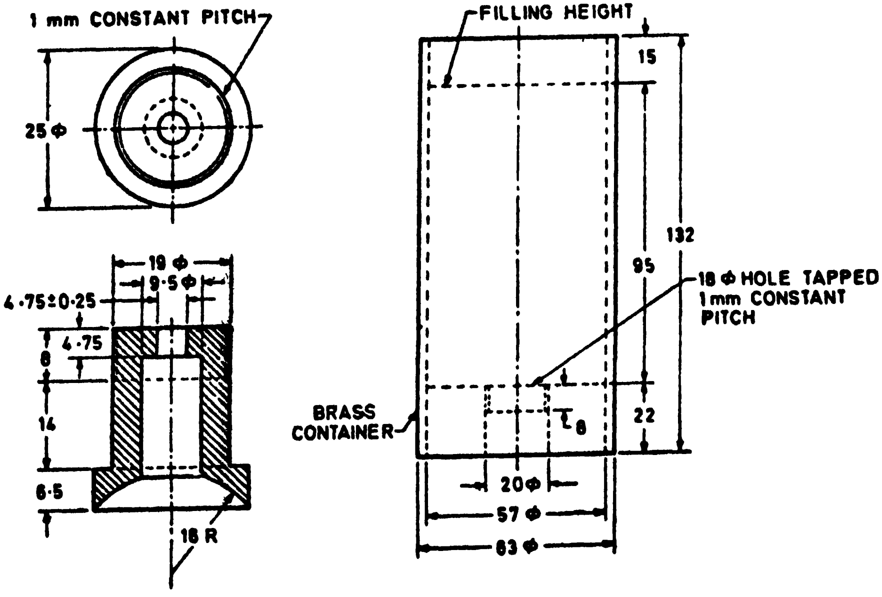
A-2. APPARATUS

A-2.1 The apparatus for the determination of the pouring point of the compound is shown in Fig. 1 to 5. The container for holding the compound, the jet and the valve have the dimensions shown in Fig. 1 and 2. Details of the apparatus, arranged for electrical heating, are shown in Fig. 3 and 4. If gas heating is to be used, the container shown in Fig. 1 is fitted into a thick-walled copper vessel into which two copper lugs are screwed and brazed (*see* Fig. 5). With either form of apparatus the container should be a close fit in the outer vessel. The construction of the apparatus is described in **A-2.2** to **A-2.4**.

A-2.2 The container in which the compound is placed consists of a brass tube having a wall thickness of approximately 3 mm, silver soldered into a massive brass block 22 mm thick, as shown in Fig. 1. The jet is of case-hardened steel, as shown in Fig. 1A.

A-2.3 A pin-type valve of silver-steel rod of 6·3 mm diameter (*see* Fig. 2) with a fitting to hold the thermometer, closes the orifice. The valve is fitted with a short pin so that it may be supported in the ring-holder above the orifice during a test. The stirrer consists of a spiral of 1·6 mm diameter spring steel wire (*see* Fig. 2). The receiver is a transparent silica flask graduated at 50 ml intervals (*see* Fig. 3). (A silica or borosilicate glass test-tube 22 mm in diameter may also be used.)

A-2.4 The container has been a mark cut on the inside all round the tube at a distance of 95 mm from the upper surface of the brass block. When electrical heating is to be used, the container fits closely into an outer vessel of brass mounted on a brass block (*see* Fig. 3). The external surface of the vessel is insulated with several layers of asbestos paper on which is wound nickel-chromium wire kept in place with refractory cement (*see* Fig. 4). As thermal insulation, several layers of asbestos paper are

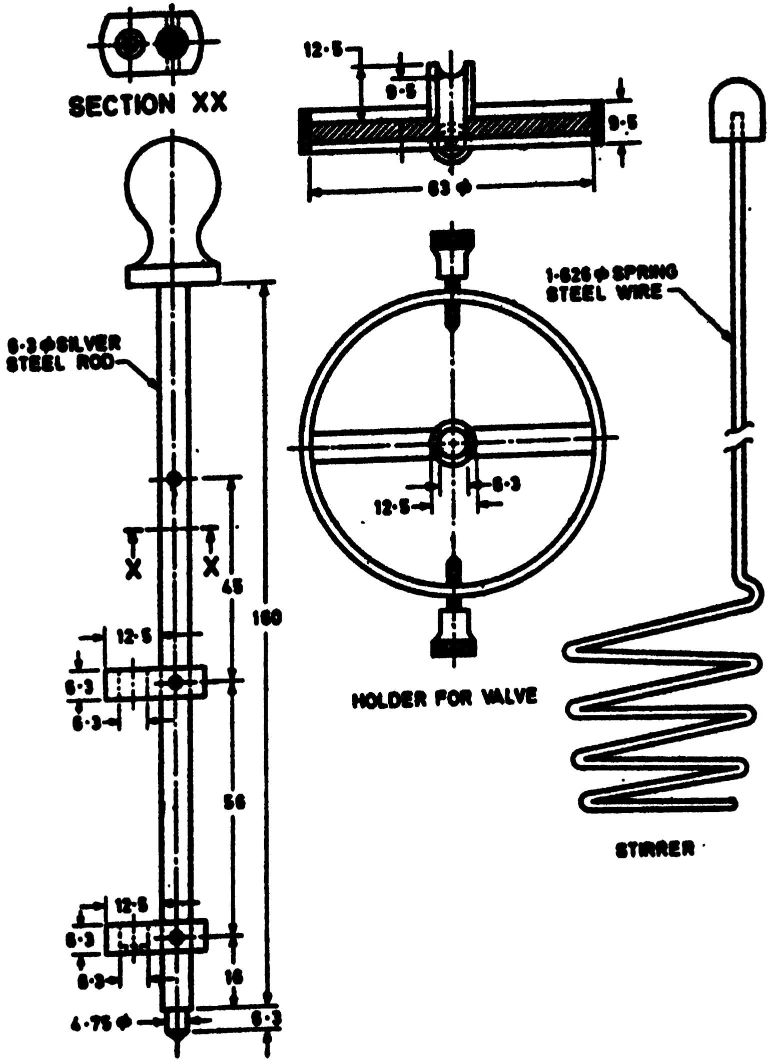


1A Jet

1B Container

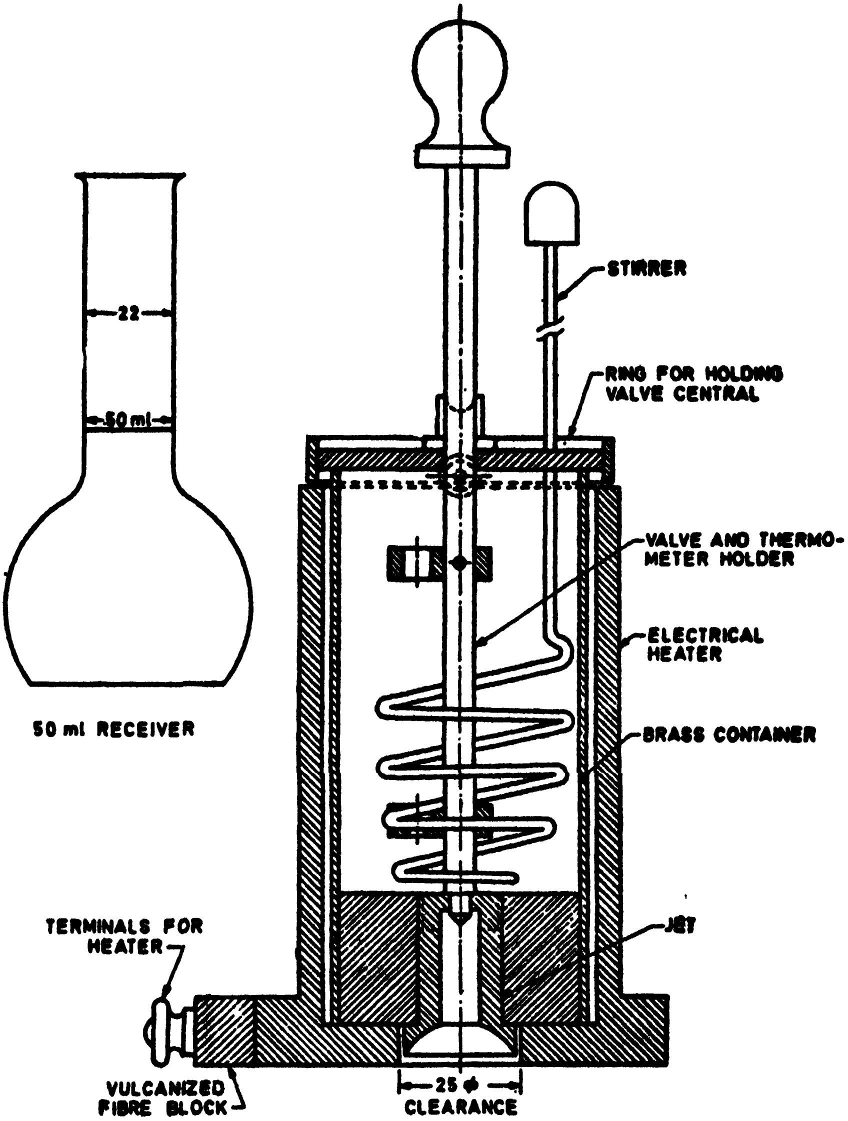
All dimensions in millimetres.

Fig. 1 Container for Compound and Jet



All dimensions in millimetres.

Fig. 2 Valve, Holder and Stirrer



All dimensions in millimetres.

FIG. 3 APPARATUS FOR DETERMINATION OF POURING POINT ARRANGED FOR ELECTRICAL HEATING

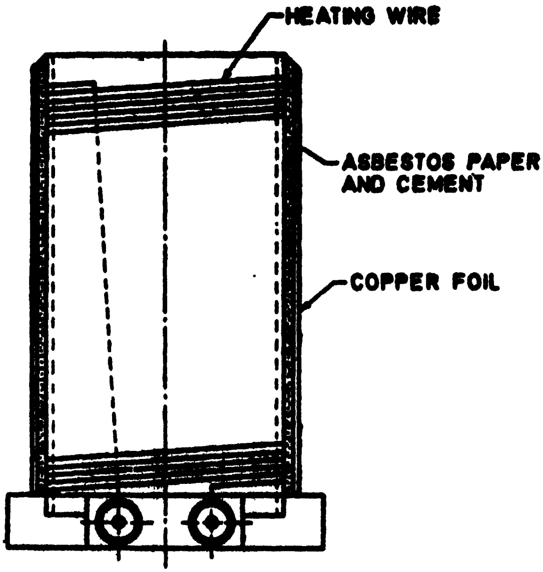


FIG. 4 OUTER VESSEL FOR ELECTRICAL HEATING

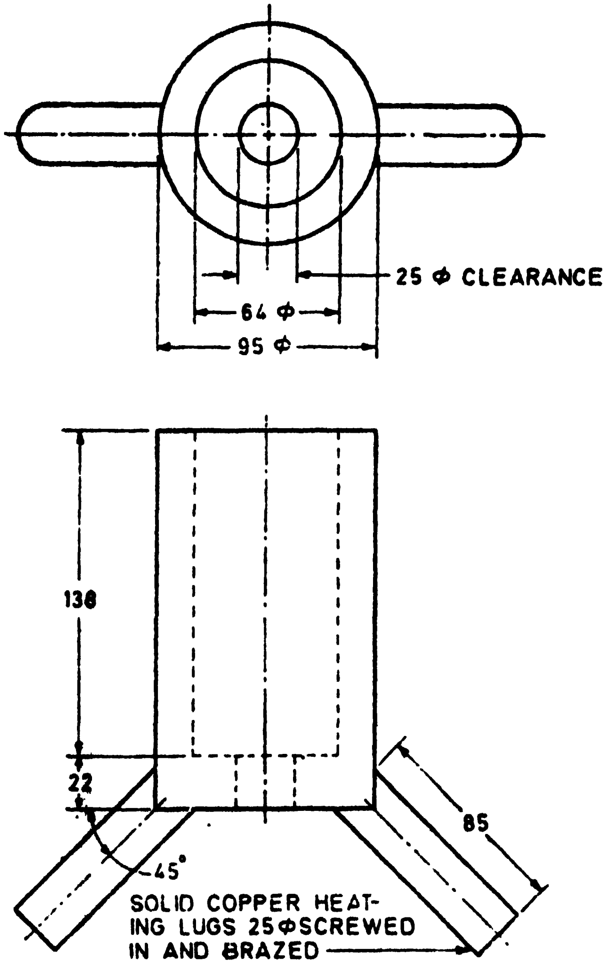
wound over the heater and covered with a copper foil sheath. For temperature control, a suitable variable resistor is connected in series with the heater. For testing the compounds covered by this standard the heater shall be suitable for dissipating not less than 200 watts.

NOTE — The size and number of turns of wire depend on the voltage of the circuit to which the heater is to be connected. For a supply between 200 and 230 volts, 100 turns of 0·315 mm diameter nickel-chromium wire is suitable for testing the compounds covered by this standard.

A-2.5 When gas heating is used, the outer vessel is turned out of solid copper and fitted with two copper rods (heater lugs) screwed and brazed into the vessel (*see* Fig. 5). In other respects the apparatus is similar to that described in **A-2.2** to **A-2.4**. The brass container for holding the compound should be a close fit in the outer vessel.

A-3. PROCEDURE

A-3.1 The compound is heated to a temperature 90-100°C above the softening point (*see* **4**) and poured into the container (previously placed in the outer vessel and raised to high temperature) up to the 'filling mark'. When the temperature of the compound (with continuous stirring) has been steady for about 2 minutes, the pin-valve is raised and supported in the holder clear of the jet, and the compound flows out into the graduated



All dimensions in millimetres.

FIG. 5 OUTER VESSEL FOR GAS HEATING

receiver (*see* Fig. 3). A stop-watch, reading to one-fifth of a second, is used for determination of the time required for 50 ml of compound to flow out. The stop watch is wound up fully before each test.

A-3.2 To ascertain the pouring point of a compound not less than four tests are made at different temperature, and the time of out-flow of 50 ml of compound is determined in each case. The tests are made with ascending values of temperature up to a temperature at which 50 ml of compound flows out in approximately 10 seconds. To avoid decomposition, care should be taken that the compound is not overheated; this may be recognized by dense or coloured fumes or by the formation of a skin on the surface of the compound.

A-3.3 To determine the pouring point, the results of the tests are plotted with the temperatures in degrees Celsius against the logarithms (base 10) of the times of out-flow in seconds. The pouring point is then obtained by interpolation from this curve.

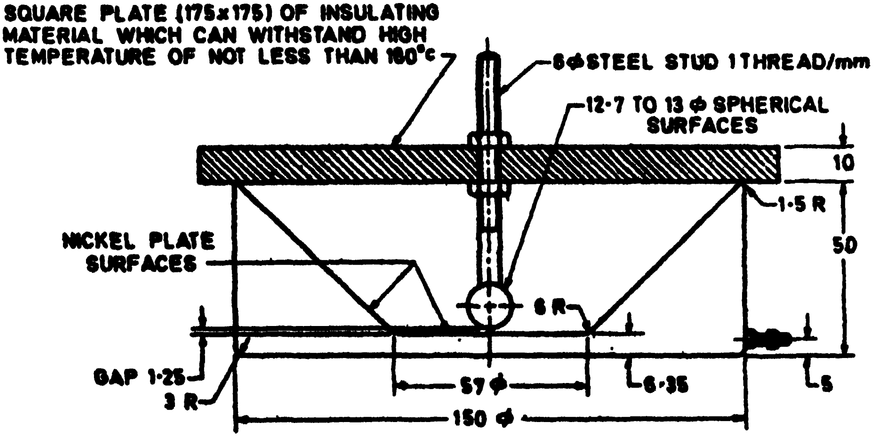
APPENDIX B

(*Clause* 8.1)

ELECTRIC STRENGTH (PROOF) TEST

B-1. APPARATUS

B-1.1 A suitable form of container is a steel cup, the inside surface of which is thickly nickel plated and having the dimensions shown in Fig. 6. A satisfactory method of mounting the electrode is also shown in the figure.


SQUARE PLATE (175×175) OF INSULATING MATERIAL WHICH CAN WITHSTAND HIGH TEMPERATURE OF NOT LESS THAN 160°C
6ϕ STEEL STUD 1 THREAD/mm
12·7 TO 13 ϕ SPHERICAL SURFACES
10
1·5 R
50
NICKEL PLATE SURFACES
6 R
GAP 1·25
3 R
57 ϕ
6·35
5
150 ϕ

All dimensions in millimetres.

FIG. 6 APPARATUS FOR ELECTRIC STRENGTH (PROOF) TEST

B-1.2 The standard gap of 1·25 mm is set at 27 ± 2°C by means of a gauge placed in the bottom of the cup and the spherical electrode screwed down until contact is made. This may be checked by any suitable continuity checking meter. No allowance should be made for expansion of steel cup and spherical electrode assembly when testing at the appropriate temperature.

B-2. PROCEDURE

B-2.1 One spherical steel or brass electrode of diameter 13 mm is arranged at the centre, vertically above the inner flat base of a steel cup in which the compound may be poured, such that a gap of 1·25 mm exists between the sphere and the inner flat surface.

B-2.2 The compound, container and electrodes are heated to the pouring point of the compound under test.

B-2.3 The compound is then poured into the container until the spherical electrode is covered to a depth of not less than 25 mm. The vessel and compound are maintained at the pouring point for at least one hour to ensure that all air bubbles have dispersed. The compound is then cooled slowly to the test temperature. This temperature is maintained for at least three hours before the test is made to ensure that the temperature at the electrodes is the correct test temperature. To ensure that it is dry when poured, Class I compound should be poured at a temperature of not less than 100°C.

B-2.4 The electric strength (proof) test is made at the appropriate temperature given in Table 2.

B-2.5 The ac test voltage is provided by a transformer and is alternating, at any convenient frequency between 40 to 60 Hz. It should be of approximately sine-wave form with a peak factor within the limits of $\sqrt{2} \pm 5$ percent (1·34 – 1·48) at the test voltage.

B-2.6 The peak value of the test voltage may be measured by a peak voltmeter, in which case the rms value for the purpose of this standard is considered as the peak value so determined by $\sqrt{2}$.

B-2.7 Alternatively, the test voltage may be determined by a voltmeter connected to the input or output side of the testing transformer and calibrated against a sphere gap connected to the output terminals of the transformer (*see* IS : 2071-1962*).

B-2.8 The impedance of the testing set is such that the short-circuit current is not less than 20 mA at all required voltage settings above 15 kV. To prevent damage to the electrodes, the short-circuit current may be limited, if desired, by the addition of external impedance, but should not be reduced below the value stated above.

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*Methods of high voltage testing.

B-2.9 One pole of the testing transformer may be earthed. The steel cup may also be earthed during test.

B-2.10 The voltage applied to the spherical electrode is raised gradually from zero to the appropriate value given in Table 2, in approximately 10 seconds, and is maintained at that value for one minute.

APPENDIX C

(*Clause* 9.1)

DETERMINATION OF FLASH POINT (OPEN)

C-1. METHOD A

C-1.1 Procedure

C-1.1.1 The compound shall be melted at as low a temperature as possible and poured into a tin of the dimensions shown in Fig. 7 so that the level of the compound is approximately 9 mm below the top of the tin. The tin shall be heated in an air-bath of the dimensions shown in Fig. 7, the temperature being measured by a thermometer, the bulb of which is immersed in the compound. The ends of two platinum wires should be set 1·5 mm apart to form a spark gap and are fixed approximately 1·5 mm above the surface of the compound. A convenient method of supporting the platinum wire is shown in Fig. 7. The wires should be connected through a tapping key to apparatus suitable for the production of a spark across the gap when the key is closed.

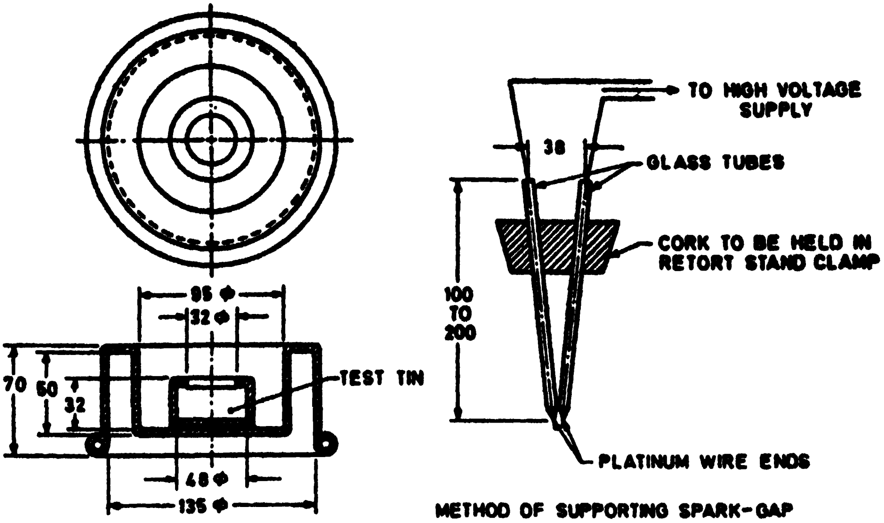
C-1.1.2 The temperature of the compound shall be raised at the rate of approximately 20°C per minute until a temperature of 20°C below the specified minimum flash point is reached. The rate of increase of temperature shall be then reduced to 5°C per minute, and during this period the taping key is closed momentarily at intervals of approximately one second until the flash point of the compound is reached.

C-1.1.3 Warning of the approach of the flash point is given by alteration in colour of the spark, which, starting from blue becomes whiter and brighter as the flash point is approached. At this stage careful observation of the rise of temperature should be made so that when the flash occurs the temperature of the compound is known accurately.

C-2. METHOD B

C-2.1 Apparatus — The apparatus consists of the following.

C-2.1.1 *Cleveland Flash Cup* — A cleveland open cup, made of brass and conforming to the dimensions shown in Fig. 8. The bevelled edge of the



HOT-AIR BATH

METHOD OF SUPPORTING SPARK-GAP

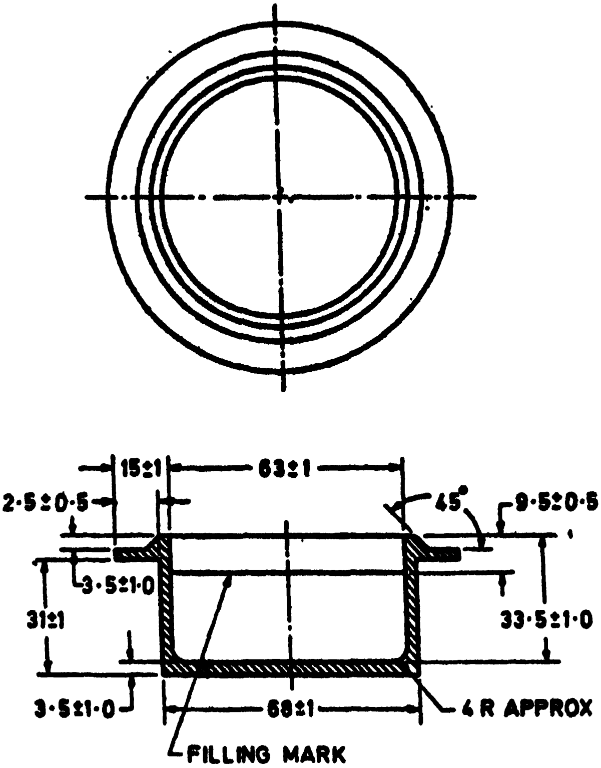
All dimensions in millimetres.

FIG. 7 APPARATUS FOR DETERMINATION OF FLASH POINT

cup is at an angle of approximately 45°. There may be a fillet of approximately 4 mm radius inside the bottom of the cup.

C-2.1.2 *Heating Plate* — This is a metal plate (*see* Fig. 9) 6 mm in thickness, and 152 mm in width, for supporting the flash cup. The plate may be of brass, cast iron, wrought iron, or steel. In the centre of the plate there is a plane depression 0·8 mm in depth, and of just sufficient diameter to fit the cup. There is a circular opening 55·5 mm in diameter, cut through the plate, concentric with the depression. The plate is covered with a sheet of hard asbestos board of 6·3 mm in thickness, and of the same shape as the metal plate. Cut in the centre of the asbestos board is a circular hole just fitting the cup. Heat may be supplied from any convenient source. The use of a gas burner, electric heater, or alcohol lamp is permitted, but under no circumstances should the products of combustion or the free flame be allowed to come up around the cup. The source of heat is centred under the opening in the plate and should be of a type that will not produce local super heating. If a flame heater is used, it may be protected from the draughts by any suitable type of shield that does not project above the level of the upper surface of the asbestos board.

NOTE — It is not necessary to use supplementary protection such as the use of a gauge between the source of heat and the bottom of the cup.



All dimensions in millimetres.

FIG. 8 CLEVELAND OPEN CUP

C-2.1.3 *Thermometer* — The thermometer shall comply with the requirements of schedule mark 12 of IS : 2480-1964*. The thermometer is suspended or held in a vertical position by suitable device. The bottom of the bulb is 6·3 mm from the bottom of the cup and half-way between the centre and wall of the cup.

C-2.2 Procedure

C-2.2.1 The compound shall be heated to a temperature of not more than 175°C to render it sufficiently fluid to pour. The cup is then filled with the sample so that the top of the meniscus is exactly at the filling line at room temperature. The surface of the sample should be free from bubbles. There should be no sample above the filling line or on the outside of the apparatus.

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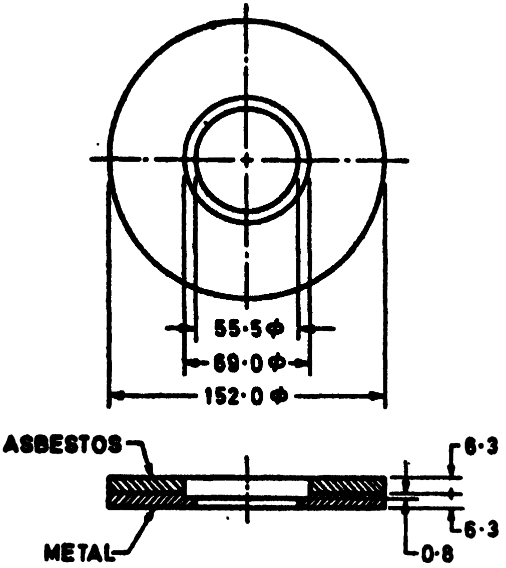
*Specification for general purpose glass thermometers.



All dimensions in millimetres.

FIG. 9 HEATING PLATE

C-2.2.2 The test flame shall be approximately 4 mm in diameter. For purposes of comparison it is recommended that a bead of suitable light coloured material be mounted in a convenient position, so that the size of the test flame may be correctly adjusted. The device for applying the flame may be of any suitable type, but it is suggested that the tip be approximately 1·6 mm in diameter at the end and that the orifice by 0·8 mm diameter. If the device for operating the test flame is mounted in such a manner as to permit automatic duplication of the sweep of the test flame, the radius of swing shall be not less than 152 mm.

C-2.2.3 The test flame shall be applied as the temperature reading reaches each successive 0·5°C mark. The flame should pass in a straight line across the centre of the cup and at right angles to the diameter passing through the thermometer. The test flame should, while passing across the surface of the sample, be in the plane of the upper edge of the cup. The time for the passage of the test flame across the cup should be approximately 1 second. The operator should avoid breathing over the surface of the sample. The test should preferably be carried out in a room free from air currents and sufficiently darkened for the flash to be readily noticeable.

C-2.2.4 The sample shall be so heated that the rise in temperature does not exceed 15°C per minute, till a point is reached approximately 55°C below the probable flash point of the sample. Thereafter the rate is decreased and for at least the last 25°C before the flash point is reached, should be neither less than 5°C nor more than 5·5°C per minute.

C-3. RESULT

C-3.1 The temperature reading on the thermometer when a flash first appears at any point on the surface of the compound shall be reported as the flash point. The bluish halo that sometimes surrounds the test flame shall not be confused with the true flash.

C-4. PRECISION

C-4.1 Results of duplicate tests should not differ by more than the following amounts:

Repeatability (One Operator and Apparatus)	*Reproducibility* (Different Operators and Apparatus)
5°C	5°C

APPENDIX D

(*Clause* 10.1)

DETERMINATION OF MINERAL MATTER (ASH)

D-1. INTRODUCTION

D-1.1 The ash content of a bituminous compound is the percentage by mass of inorganic residue left after ignition of the compound.

D-2. OUTLINE OF METHOD

D-2.1 In Method A a weighed amount of the sample is gently heated until it will burn and the residue ignited until it is free from carbon. Alcohol or a mixture of alcohol and benzene may be added to prevent the foaming of wet samples. In Method B the partially ignited residue is treated with dilute sulphuric acid and the sulphated ash weighed.

D-3. APPARATUS

D-3.1 Dish or squat-form crucible of silica, porcelain or platinum, and of not more than 25 ml capacity.

NOTE — A platinum dish should not be used if the sample contains lead, zinc, or other metals which attack platinum at high temperatures. Similarly, a porcelain dish is attacked by lead and vanadium.

D-4. METHOD A FOR COMPOUNDS OF CLASSES I TO V

D-4.1 Procedure

D-4.1.1 Heat the dish to redness, allow it to cool in a desiccator, and weigh it to the nearest 0·1 g. Weigh a small beaker containing the sample to the nearest 0·1 g, transfer successive portions of the sample to the dish and ignite them until a sufficient quantity has been used. Reweigh the beaker to the nearest 0·1 g and obtain the mass of sample used by difference. For ashes of 0·02 to 0·2 percent, take about 20 g of sample; for samples outside this range, take the proportionately larger or smaller quantity.

D-4.1.2 Heat the dish gently until the sample is ignited at the surface. Allow the combustible matter to burn off slowly and heat the residue strongly with a flame, or in a muffle furnace, until the ash is free from carbon. Cool the dish and its contents in a desiccator and weigh them to the nearest 0·1 g. Repeat the heating until constant mass is obtained.

D-4.2 Calculation and Reporting — Calculate the mass of the residue as a percentage of the original sample, and report it to two significant figures, stating also the mass of sample used.

D-4.3 Precision — Results of duplicate tests should not differ by more than the following amounts:

Repeatability (One Operator and Apparatus)	*Reproducibility* (Different Operators and Apparatus)
20 percent of mean	20 percent of mean

D-5. METHOD B FOR CLASS VI COMPOUND

D-5.1 Reagents

D-5.1.1 *Sulphuric Acid* — 10 percent approximately.

D-5.1.2 *Methyl Orange Solution* — 0·1 percent aqueous.

D-5.1.3 *Ammonium Carbonate* — solid.

D-5.2 Procedure — Burn the sample as described in Method A (*see* **D-4.1**), until the ash is nearly free from carbon. Cool the dish and its contents, dissolve the soluble portions in a little water and add carefully a slight excess of sulphuric acid from a pipette inserted under a watch-glass covering the dish. Warm the dish and its contents on a boiling water-bath until effervescence has ceased, and then rinse the watch-glass with water into the dish. Test the solution with methyl orange to ensure the presence of free acid. Evaporate the contents of the dish to dryness and ignite them at a low red heat, adding a few small pieces of dry ammonium carbonate to drive off the excess of sulphur trioxide. Cool the dish and contents in a desiccator and weigh them.

D-5.3 Calculation and Reporting — Calculate and report the mass of the residue as a percentage of the original sample to the nearest 0·1 percent.

APPENDIX E

(*Clause* 11.1)

DETERMINATION OF INJURIOUS SULPHUR

E-1. PROCEDURE

E-1.1 Separate about 1 g of the compound into pieces as small as practicable and place in a porcelain dish. About 50 ml of a suitable solvent (for example, chloroform, carbon tetrachloride, methylene chloride) shall be added and the dish is then heated gently on a water-bath. A piece of freshly polished copper foil approximately 50 mm square is cleaned and polished with a pad of cotton wool and a fine abrasive powder and rubbed with successive pads of cotton wool until a fresh pad remains unsoiled after having been rubbed on the foil. Completely immerse the foil in the warm solution for half an hour, the bulk of the liquid being maintained by occasional additions of the solvent as required. On removal of the copper foil from the solution it should be washed with the liquid used as the solvent and examined for discoloration.

E-1.2 The solvent used should be such that the blank test carried out as described above will not discolour the copper.

E-2. RESULT

E-2.1 The result is reported in accordance with the following colour scale:

a) No change, and

b) Slight discoloration.

APPENDIX F

(*Clause* 12.1)

DETERMINATION OF ACIDITY

F-1. INTRODUCTION

F-1.1 This method gives a measure of the total acidity, that is, the combined organic and inorganic acidity, of the compound.

F-2. OUTLINE OF METHOD

F-2.1 The compound is dissolved in a mixture of benzene, alcohol and water, a slight excess of alkali added, and the mixture back-titrated with acid.

F-3. APPARATUS

F-3.1 Titration flask of 250 ml capacity, with ground glass stopper, and with a long, narrow neck or a volumetric flask may be used.

F-4. REAGENTS

F-4.1 Potassium Hydroxide Solution — 0·1 N, accurately standardized. Prepare this solution with freshly boiled and cooled distilled water, and store it in a bottle fitted with a guard-tube of soda lime to prevent access of carbon dioxide. Restandardize the solution at frequent intervals.

F-4.2 Hydrochloric Acid — 0·1 N, accurately standardized.

F-4.3 Alkali Blue Solution — Extract 2 g of alkali blue 6B with boiling alcohol by means of a Soxhlet apparatus, filter it if necessary, and dilute the solution to 100 ml with alcohol.

F-4.4 Benzene — conforming to IS : 1840-1961*.

F-4.5 Ethyl Alcohol — Dilute 85 parts of alcohol with 15 parts of water, by volume.

F-5. PROCEDURE

F-5.1 Weigh about 5 g of the sample to the nearest 0·01 g into a 250-ml conical flask. Dissolve the sample in about 60 ml of benzene by heating and swirling, and transfer the hot solution quantitatively to the titration flask. To 160 ml of the dilute alcohol add 20 ml of the alkali blue solution and one drop of the hydrochloric acid, and neutralize the mixture with the potassium hydroxide solution; add this mixture to the solution of the sample. Titrate the sample with the potassium hydroxide solution, shaking the flask vigorously after each addition of alkali, and add an excess of about 0·5 ml of alkali. Back titrate with the hydrochloric acid.

NOTE — The colour change, which is clearer when titrating back with acid than in direct titration, may readily be observed in the thin layer of liquid in the narrow neck of the flask.

F-5.2 Make a blank determination in the same way, but omitting the sample.

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*Specification for benzene, reagent grade.

F-6. CALCULATION AND REPORTING

F-6.1 Calculate the acidity of the bitumen from the formula:

$$A = 56\cdot1\,[\,(V - V_1)\,N - (v - v_1)n\,]/M$$

where

A = acidity in mg KOH/g,
V = volume in millilitres of alkali used in the determination,
V_1 = volume in millilitres of alkali used in the blank,
N = normality of the alkali,
v = volume in millilitres of acid used in the determination,
v_1 = volume in millilitres of acid used in the blank,
n = normality of the acid, and
M = mass in grams of sample.

F-6.2 Report the result to the nearest 0·1 mg KOH/g as the acidity.

F-7. PRECISION

F-7.1 Results of duplicate tests should not differ by more than the following amounts:

Repeatability (One Operator and Apparatus)	*Reproducibility* (Different Operators and Apparatus)
0·2	0·2

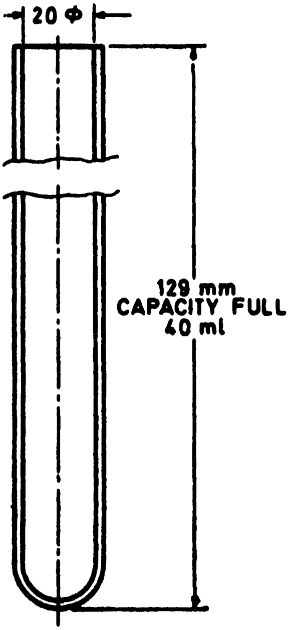
APPENDIX G

(*Clauses* 13.1 *and* 13.2)

METHOD OF TESTING CONTRACTION AND DEPTH OF PIPE

G-1. PROCEDURE

G-1.1 Contraction and depth of pipe are measured by using a glass tube of 40 ml nominal capacity and 20 mm internal diameter, having flat ground edges (*see* Fig. 10). The tube is heated to 150°C, slightly overfilled with the compound under test, also preheated to this temperature, and maintained at 150 ± 1°C for one hour when compound of Classes I, II, III or IV is being tested and for two hours when compound of Classes V or VI is being tested. At the end of this period the compound is levelled to the top of the tube by means of a heated metal spatula. The compound filled tube is then immediately transferred to the laboratory bench and cooled to a temperatue of 20 ± 1°C. In order to control the rate of cooling the tube is enclosed throughout this period in a draught screen. This screen is 225 to 250 mm high and may be a cylinder of 150 to 175 mm diameter, or a rectangular screen having 150 to 175 mm sides; the top is covered. If the laboratory temperature exceeds 21°C, after cooling in air to the laboratory temperature, the tube is immersed in a water-bath and cooled to a temperature of 20 ± 1°C. The temperature measurements are taken with a thermometer immersed in another similar tube of compound, which is submitted simultaneously to the same heating and cooling cycle as the compound under test.



All dimensions in millimetres.

FIG. 10 GLASS TUBE FOR CONTRACTION AND DEPTH OF PIPE

G-2. CONTRACTION

G-2.1 Care should be taken to ensure that the compound is free from internal cavities after cooling to 20°C and that the pipe formed in the compound as the result of cooling it in air to 20°C is filled with methylated spirit from a burette. The volume of spirit required to fill the pipe is noted. From the figure obtained the percentage contraction is calculated.

G-3. DEPTH OF PIPE

G-3.1 The depth between the ground mouth of the tube and the bottom of the pipe in the compound at 20°C is measured by means of a depth gauge or other suitable instrument.

APPENDIX H

(*Clause* 15.1)

METHOD OF TEST FOR ADHESIVENESS

H-1. APPARATUS

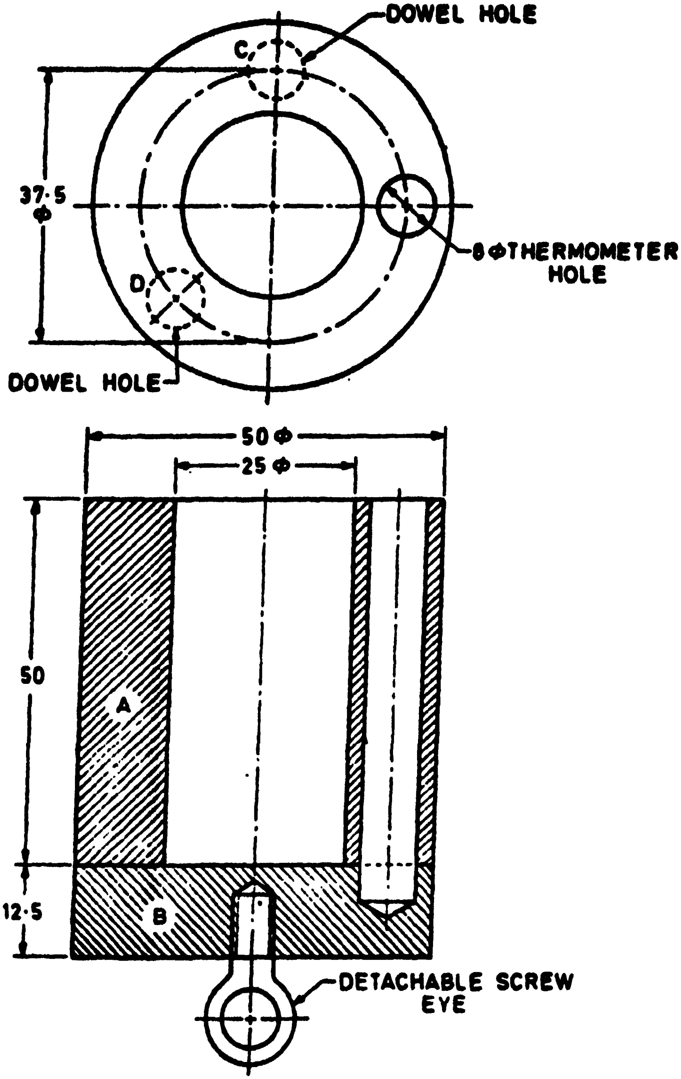
H-1.1 A steel mould (*A*) in form of a cylinder of the dimensions shown in Fig. 11 rests on a steel plate (*B*). The mould contains two dowels (not shown) fitted in one end, which register easily in holes (*C*) and (*D*) in the plate. A hole is drilled through the wall of the mould and continued into the plate to receive a thermometer. The upper surface of the steel plate (*B*) is polished as explained in **H-1.2**.

H-1.2 The surface of the steel plate is prepared either by means of a recognized polishing machine as used for the preparation of specimens for metallographic examination, or by hand with a fresh clean sheet of No. 1 emery cloth. In the latter method the emery cloth is placed on a flat surface and the steel plate laid face downwards on the emery cloth. The plate is rubbed in one direction (to and fro), a circular motion being avoided, until all marks are in that direction, and then in a direction at right angles until all marks are in the latter direction. The double rubbing is done not less than three times and until inspection of the surface of the plate shows that all the scratches are new.

H-2. PROCEDURE

H-2.1 Immediately after cleaning as described above, the apparatus is placed in an oven until the thermometer registers the appropriate temperature specified in Table 4. The compound under test, having been heated to the pouring point, is poured into the mould until the latter is full.

NOTE — When the pouring point is not known, the compound may usually be heated safely to 80°C above the softening point.



All dimensions in millimetres.

FIG. 11 APPARATUS FOR DETERMINATION OF ADHESIVENESS

H-2.2 The apparatus and compound are allowed to cool to a temperature of 27 ± 5°C and are maintained at this temperature for not less than one hour. A ring or other form of handle is screwed into the underside of the plate and the mould and plate are subjected to a slow steady pull until they are separated. The plate is examined and the percentage area of adhesion reported. A brown stain is not regarded as an indication of adhesion between the compound and the base plate.

TABLE 4 MOULD TEMPERATURES

(*Clause* H-2.1)

CLASS	MOULD TEMPERATURE
I	—
II	Softening point temperature
III	Softening point temperature
IV	20°C above softening point
V	40°C above softening point
VI	Softening point

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